

FLUIDIZATION CONTROL IN THE WURSTER COATING PROCESS

Particulate coating process in a fluidized bed involves different subprocesses including particle wetting, spreading and also consolidation or drying of the coating applied. These subprocesses are done simultaneously to particle fluidization and motion. All the parameters of fluidization are known to affect the coating quality. That is why the motion of particles in the Wurster coating process has been observed and described step by step. These observations have achieved a general understanding of phenomena which take place inside the bed during fluidization and have allowed the development of an easy method for optimizing all the parameters affecting this operation.

Particle coating in fluidized beds is of high interest in pharmaceutical and agricultural applications to ensure the protection, controlled release and functionality of active ingredients in solid particulate form. Three different types of equipment are used in industry: pan coating, top-spray fluid-bed coating and Wurster type coating. To coat pellets, the bottom spray fluidized bed or Wurster coating process is mostly used and it is this process that will be studied in the article. The process involves wetting and drying in close combination with particle movement. This article is aimed to achieve a general and specific understanding of phenomena taking place inside the bed during fluidization and to develop a new method to optimize this operation. Both observations, experimental data and simulation tools gave a better understanding of the process which is a prerequisite to guarantee a high and stable product quality. This knowledge will subsequently be used in improved design, scale-up of coating equipment.

The principle of the fluid-bed particulate coating process is to suspend particles in a fluid-like state using gas introduced at the bottom of the bed. Depending on the position of the spraying nozzle, the coating solution is sprayed directly into or on the particles. The gas that acts as a momentum carrier improves the coating drying. Hence, the coating layer of the particles is built up during wetting and drying together with particle motion. As described in Figure 1, the Wurster equipment consists of a cylindrical insert (also called the coating partition or tube) inside an outer quasi-conical chamber with a gap between the insert and the bottom distributor plate. The open area in the distributor plate in those two sections determines the relative amount of air flowing into the insert and the annulus [1].

During coating, the solid particles are spouted in an upward-moving air stream through the insert, where they receive coating from a spray nozzle located on the distributor plate at the bottom of the insert. The particles dry as they pass upward into the top of the partition, where they lose their momentum and fall into the

annulus. The particles then flow downward in the annulus in a plug flow manner and re-enter the insert through the partition gap at the bottom of the annulus. This circulation of particles is repeated until the desired coat amount is deposited on the solid particles.

MATERIALS AND METHODS

The experimental apparatus is the Uni-Glatt pilot (Glatt GmbH, Binzen, Germany) with a draft tube. The inox draft tube was replaced by a Plexiglas tube having 0,075 m diameter and 0,15 m height.

Spherical microcrystalline cellulose particles distributed in a monodispersed population of 1.08 mm in average diameter were used as the circulating medium in the experiment. The particle density was $1356 \text{ kg}\cdot\text{m}^{-3}$. Air at ambient temperature was used as the fluidizing gas.

The pressure drop was measured with and without particles using two transmitters of differential pressure (Model P92, Etoile International, France) placed between the air entrance and the top of the bed.

RESULTS AND DISCUSSION

Observation of particle motion

The Wurster process can be divided in four regions (Figure 2): (A) the upbed region in the inner cylinder, (B) the deceleration region in the expansion chamber, (C) the downbed region in the annulus and, finally, (D) the compact bed region at the base of the chamber. The size of each region is determined mainly by the dimensions of the apparatus, but also to some extent by process parameters such as the product load or batch size and air flow rate [2].

In the upbed region (A), particles are transported vertically by pneumatic conveying with a very high porosity. In this region, particles are wetted by the sprayed coating liquid and they begin to dry before reaching the second region.

Particles leave the upbed region to enter the expansion region where they are in deceleration. They travel a certain distance upwards before losing their momentum and start to fall down in the annulus. The drying of the film on the particles continues in this region.

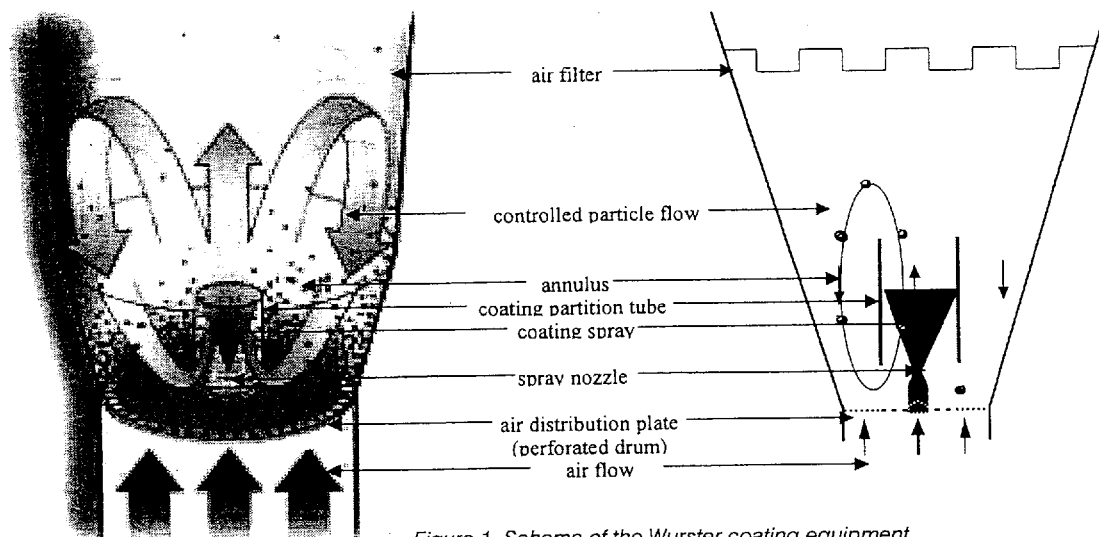


Figure 1. Scheme of the Wurster coating equipment

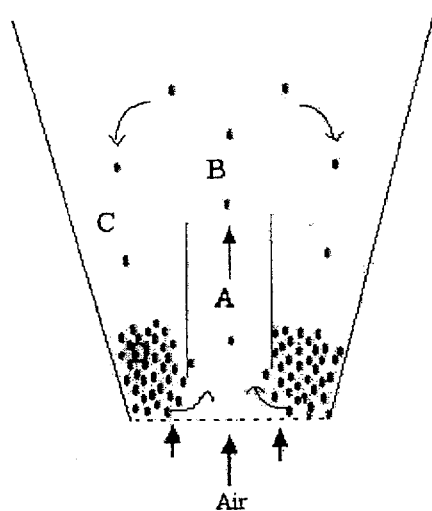


Figure 2. Particle flow regions in the Wurster process

In the downbed region (C), the particles flow downward in the annulus in a plug flow manner before they re-enter the upbed region. This region allows additional drying of the particles which must be completely dried to avoid sticking and agglomeration.

The compact bed region (D) consists of a tampon region where particles are moving slowly in successive jumps towards the upbed region. The opening under the partition of the Wurster controls this horizontal transport, which defines the solid mass flow rate per unit area in the upbed region.

The quality of the formed coating depends mainly on the airflow of fluidization. A significant speed of fluidization air improves the circulation of the particles and ensures good mass and heat transfer [3].

Method for fluidization control

To determine the optimal parameters for good fluidization, several conditions must be checked: i) the regular transport of particles, ii) good density of the particles in the insert and iii) the limitation of particle agitation in the annulus.

Currently, the method to optimize fluidization was acquired from long experience with the process or visually by controlling the density of the particles in the insert at rest. The cover of the nozzle of pulverization by the particles indicates that the selected parameters are adequate. Thus the pressure drop between the insert and the annulus must be a good indication of the density of the particles and so its record may be useful for the control of fluidization.

Figure 3 represents the pressure drops due to particles in the annular space and the central part. This pressure drop was calculated by the difference between the pressure drop measured by the particle and pressure drop due to the air plate distributor (without particles). The airflow rate was determined by the Uni-Glatt equipment.

As the airflow rate was progressively increased, the initially packed bed passed through a transition region and ultimately achieved a fully developed spouted condition. Observations of the three stages may be described as follows:

At low airflow rate, the air passed through the bed without disturbing the solid particles the pressure drop increased linearly with the airflow rate. When the air rate was increased to a certain value, some of particles started to move upward in the center from the air entrance that confirmed the little difference in the pressure drop between the annulus and inside the insert.

When the air rate increased to a certain value, the particles moved up through the spout and fell through the annulus like a fountain. The pressure drop in the insert decreased suddenly and remained constant during the spouting. The particles were then in the equilibrium state and were transported in a regular way.

The optimal air flow rate is just the point after the spouting point S (Figure 3) where the circulation of the particles seems to be regular. In order to confirm this hypothesis, the evolution of the pressure drop through the annulus and through the insert was recorded continuously for each airflow rate (Figure 4).

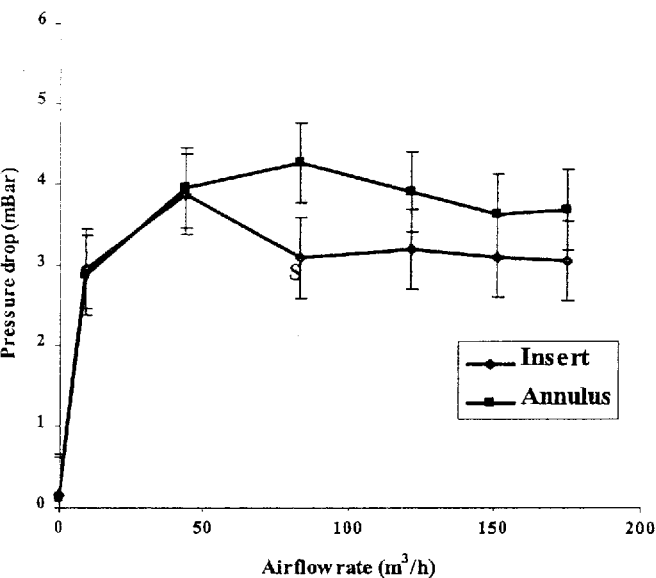


Figure 3. Pressure drop in the insert and the annulus according to the airflow rate

The pressure drop in the annulus can be divided in four steps: S2 and S4 are characterized by important pressure drop fluctuations and S1 and S3 by a stable pressure drop.

State 1 refers to the packed bed, so that the pressure drop in the annulus is stable. By increasing the airflow rate, some particles started to move up the insert since others are entrained from the annulus but not at the same speed. Therefore, we observed some fluctuations in the pressure drop (state 2). At a determined airflow rate, the particles were entrained from the annulus to the upbed region at the same speed of other particles moving up the annulus (regular transport) so that the pressure drop did not vary with time (state 3), which corresponded to the optimal conditions of fluidization. A further increase in the gas flow rate (state 4) caused vigorous transport in the annulus referring to the fluctuations in the pressure drop.

Method to optimize the batch size or inventory

Fluidization is affected by the quantity of particles which are introduced into the coating chamber: at least 50% of the volume external to the partition or the Wurster tube must be occupied by particles to be coated [4]. This makes it possible to have a sufficient quantity of particles inside the partition to accumulate the maximum coating solution droplets and to avoid thereafter the phenomenon of premature drying or depositing on the walls of the partition.

To calculate the load of particles to be introduced, it is enough to calculate the external total volume of the partition, to multiply it by the density of the particles in bulk which gives the total capacity in kg as described in the equation:

$$M = (r_1^2 - r_2^2) \pi L \rho_p$$

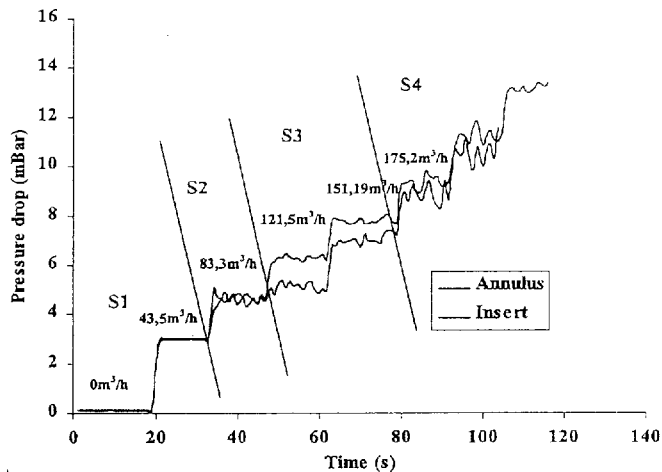


Figure 4. Pressure drop in the insert and the annulus for each airflow rate as a function of time

were: r_1 and r_2 are, respectively, the chamber radius and the partition radius, L is the length of the partition, ρ_p is bulk the density of particles.

In the case of a multi-partition process, the volume of partition must be multiplied by the number of partitions.

The table below represents the calculated efficiency of the operation of coating of microcrystalline cellulose particles (MCC) with gum Arabic for two different inventories: 250 g corresponding to 20% of the maximum capacity and 820 g (50% of the maximum capacity).

Table 1. Efficiency calculated according to batch size

Particles	Coating material	Batch size (g)	Efficiency (%)
MCC (850-1000 μ m)	Gum acacia	250	63%
MCC (850-1000 μ m)	Gum acacia	800	98%

At least half of this capacity will be enough to ensure that there is sufficient material in the up bed region (insert) to accumulate all or most of the coating material being sprayed. If the product in the annulus "down bed" and its depth is insufficient, the up bed will be sparse, favoring spray drying or coating of the inner wall of the insert, which is the source of the poor efficiency.

Effect of partition height

The partition height can be varied in the Uni-Glatt from 5 to 20 mm. The void fraction ε was calculated for three various partition heights (Figure 5) by the relation $\varepsilon = 1 - (\Delta P / (\rho_p \cdot g \cdot H))$ where ΔP , ρ_p , g and H represent, respectively, the pressure drop in the insert, the particle density, the gravity acceleration and the distance between the two pressure transmitters.

The concentration of particles inside the insert was also calculated using the relation $C_p = (1 - \varepsilon) \cdot 100$ where ε is the void fraction. Figure 6 represents the concentration of particles inside the insert according the partition height.

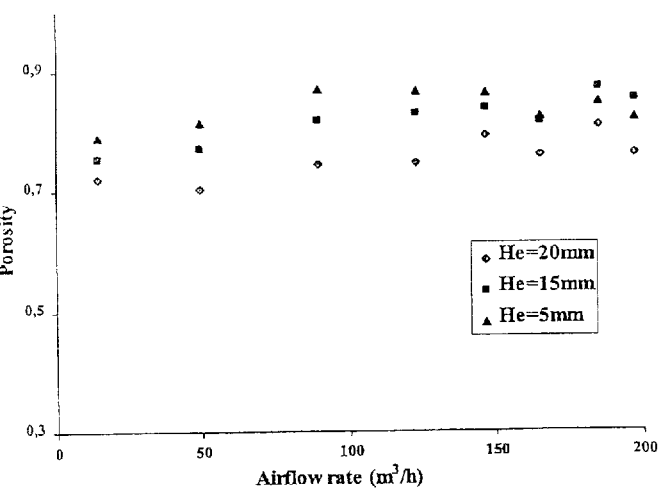


Figure 5. Void fraction according to airflow rate for different partition heights (He)

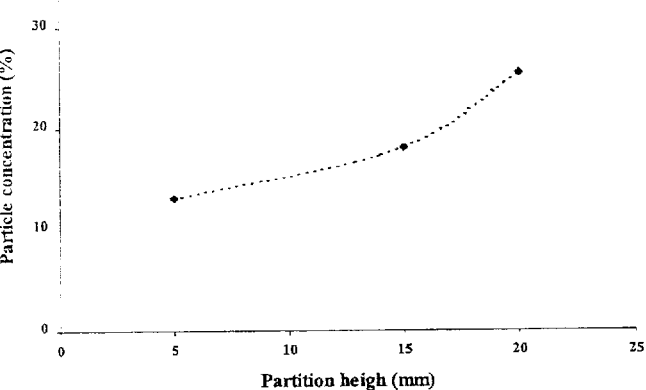


Figure 6. Particle concentrations in the insert according to partition height

As the partition height increases, the void fraction in the insert decreases slightly. This means that the partition height affects the transport of particles from the annulus to the insert. In fact, the concentration of particles inside the insert increases because of the enlargement of the entrainment zone when increasing

the partition height. This can avoid the phenomena of spray drying and improves the efficiency of the coating operation.

CONCLUSION

In the Wurster coating process, fluidization is one of the most significant operations. Its control seems a peremptory necessity before beginning the process of pulverization and drying. The parameters controlling fluidization consist of the inventory or the load in the particles, the airflow of fluidization and the partition height. By controlling these three parameters, a good circulation of the particles is ensured and a good support to begin pulverization and drying is prepared. This control can be done by measuring the pressure drop inside the process. This can be a very useful method to determine the optimal conditions of fluidization and also to ensure the quality of the coating in fluidized bed operation.

NOTATION

Variables	Units
C_p – Particle concentration	%
g – Gravity coefficient	m/s^2
H – Height distance	m
H_e – Partition height	m
ΔP – Pressure drop	mbar
L – Partition length	m
M – Total mass capacity	kg
r_1 – Chamber radius	m
r_2 – Partition radius	m
ϵ – Void fraction	–
ρ_p – Particle density	$kg \cdot m^{-3}$

REFERENCES

- [1] D.E. Wurster, Air suspension technique of coating drug particles, *Journal of American Pharmaceutical Association* (1959), 451–463
- [2] F.N. Christensen, Bertelsen, P., Qualitative description of the Wurster based fluid bed coating process., *Drug Development and industrial Pharmacy*, **23** (5) (1997) 451–463.
- [3] D.M. Jones, Air suspension coating for multiparticulates, *Drug Development and industrial Pharmacy*, **20** (20) (1994) 3175–3206.
- [4] D.M. Jones, Air suspension coating, *Pharmaceutical Technology Encyclopedia* (1988a) 189–216.

IZVOD

KONTROLA FLUIDIZACIJE TOKOM OBLAGANJA ČESTICA U WURSTER PROCESU

(Naučni rad)

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Oblaganje čestica tankim filmom u fluidizovanom sloju uključuje niz sukcesivnih procesa kao što su vlaženje čestica, nanošenje tankog sloja materijala (polimera) na čestice, i očvršćivanje, odnosno sušenje nanetog materijala. Ovi procesi se dešavaju simultano sa fluidizacijom i kretanjem čestica. Uzmajući u obzir da parametri fluidizacije utiču na kvalitet oblaganja čestica, u radu je analizirano kretanje čestica u Wurster procesu i opisano korak po korak. Ova analiza je dovela do generalnog shvatanja fenomena koji se dešavaju unutar sloja tokom fluidizacije i razvoja jednostavnog modela za optimizaciju svih parametara koji utiču na ovaj proces.

Ključne reči: Fluidizacija • Wurster proces • Pad pritiska • Koncentracija čestica •

Key words: Fluidization • Wurster process • Pressure drop • Particle concentration •